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CONTAMINATION CONTROL IN SPACE SIMULATION CHAMBERS

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ABSTRACT

Protection of sensitive vehicle surfaces during space simulation tests requires that the chamber be clean. Various methods of cleaning are considered. Cleaning is a progressive process; the methods must be altered as cleanliness improves. If an inappropriate method is used, varnish residues may form. Experience with vacuum outgassing a 39 ft. diameter space chamber is reviewed. It is shown that acceptably low contamination rates may be achieved with the present state-of-the art. Possible future developments are discussed, including the use of fluorocarbon pump oil, and cleaning with atomic oxygen.

INTRODUCTION

The customers of a space simulation laboratory are frequently concerned that their space vehicles may be damaged by contamination during test (References 1 and 2). For example, the reflectivity of optical components, and the absorptance/emittance characteristics of thermal coatings can be significantly changed by material deposited during tests.

Warm material under vacuum can be transferred to the vehicle in a direct line of sight. During normal operation the cold shroud condenses and holds any material, thus preventing contamination; however, some small warm surfaces may be present: e.g., infrared arrays, ionization gage filaments, or observation windows. Also, during the chamber-pumpdown shroud-cooling operation, and during the inverse warmup and backfill operation, it is possible to transfer contaminants. It should be noted that significant transfer from one part of the space vehicle to another may also take place (References 3 and 4).

Another way for material to be deposited in the space vehicle is through abnormal operations or accidents in the roughing or diffusion pumps. These can cause massive backstreaming, even though no measurable backstreaming is usually observed during normal operation (Reference 5). Although the risk of these accidents is very low, the potential damage must be considered.

The operator of a space simulation facility is, of course, responsive to the needs of his customers. He must not only operate the facility to minimize any contamination, but also must present convincing evidence that contamination flux levels are acceptable. In addition, parts of the facility such as solar optics may be damaged by films of contaminant. Thus, the customer and the operator have a strong mutual interest in reducing contaminant flux.

Space chamber operations differ significantly from those of conventional vacuum chambers. Installation of a space vehicle is a complex mechanical operation. It creates a certain background of contamination, no matter how clean the chamber. amount and constituents of this installation contamination is an interesting subject for further study. Another difference from conventional vacuum chamber operations lies in the relative unimportance of total vacuum level and of the common outgassing constituents - HoO, No, etc. On the other hand, there is always great concern about specific contaminants, such as

pump oils, or materials from previous spacecraft.

Many approaches are used to safeguard the space vehicle. Critical areas are shielded with remotely actuated covers. A purge of clean gas may protect the vehicle; timing of purge shutoff and shroud cooldown is optimized to avoid any unprotected interval. However, to supplement these methods of protection, it is important that the chamber be clean, that the flux of contaminating molecules be acceptably low, and that the risk of contamination be acceptable even if worst-case mishaps occur.

This paper discusses the steps that may be taken by the facility operator to minimize contamination flux levels and the risks of contamination. It is desirable for the plan to be flexible enough to provide either adequate cleanliness at minimum cost, or to reach extreme cleanliness with extra operations.

The generalized approaches to cleaning a space chamber are listed in Table 1. These are arbitrarily divided into basic methods, which are those immediately available to most facilities and improved methods, which usually cannot be implemented without some special effort.

Table 1

CLEANING METHODS

BASIC METHODS - immediately available at most facilities

Washing - Sweep, Brush, Vacuum

- Water, Steam, Detergent
- Solvents

Vacuum Outgassing - Vacuum Soak

- Transient Flow Outgassing
 - Transient Flow Outgassing with gas purge
 - Molecular Flow Outgassing
 - Elevated Temperature, with any of above

IMPROVED METHODS - usually require special effort to implement

Energized Surface - Shock, Vibration, Gas Jets

- Heating RF Pulses
 - Laser

Energized Molecules - Electron Bombardment

- Ion Bombardment
- UV Radiation Solar Beam
- Chemical Reaction Atomic Oxygen

Preventive Methods - Perfluoroalkyl Polyether Fluid - Diffusion and Mechanical Pumps

- Porous Metal Screens over Pumps
- Material and Operation Selection

BASIC METHODS OF REMOVING CONTAMINANTS - WASHING AND VACUUM OURGASSING

WASHING

Washing is necessary if large quantities of contaminant are present. Vacuum outgassing a dirty chamber has been found to leave on the wall a powdery residue, which rubs off and stains clothes; this residue could be expected to be carried to a space vehicle during installation operations. Contaminants can also be converted into varnish-like polymer films by ultraviolet (solar) radiation or electron bombardment (Reference 6), or even by hot surfaces as low in temperature as 300°C. (Reference 7). Cleanup is a progressive process that must start with a relatively clean chamber.

Relative merits of washing methods will not be discussed here. However, there is an interesting question as to the amount of contaminant that can be tolerated without washing before proceeding with vacuum outgassing. Just how dirty can the chamber be before washing is required? It would appear that quantitative guidelines could be established; however, the

authors are not aware of any work on this point.

VACUUM OUTGASSING

In these methods, the contaminants are allowed to evaporate from the chamber surfaces into some level of vacuum. The vacuum serves to reduce the recondensation rate of contaminants so that the effective evaporation rate increases as compared to atmospheric pressure (Reference 8). The operation may be at room temperature, or accelerated by heating.

One problem to be considered is the risk of spreading localized contaminants throughout the chamber, especially into small passages behind shrouds and shields. This problem is more serious with low-vapor-pressure materials such as diffusion pump oils. This is another reason for carefully washing the chamber before outgassing. Although no general solutions to the problem are offered here, a number of specific small passages have been analyzed. In all these cases, it was determined that outgassing would reduce contaminant levels.

Variations of vacuum outgassing include vacuum soak, outgassing at transition flow conditions (with and without gas purge), and outgassing at molecular flow conditions.

Vacuum Soak

The simplest outgassing method is a vacuum soak. The chamber is roughed down, sealed, and left with pumps off. The advantages of this method are primarily economic: labor, equipment operation costs, and material use are all zero during the soak. The soak can be over a weekend, or for longer periods: the MDAC 39-ft-diameter chamber (S-1) was once soaked for

nine months, with excellent results, judging by the speed of subsequent pumpdowns.

Soaking does not produce the most rapid cleanup, however, it is surprisingly effective. In S-1 a nominal monolayer (10¹⁴ molecules/cm²) over all internal surfaces, when desorbed into the internal volume would raise the pressure by 4.6 x 10⁻⁵ torr. Many contaminants have vapor pressures substantially greater than this figure, so that they move towards an equilibrium where most of the material is in the gas phase. Even low-vapor-pressure materials, such as diffusion pump oils (typically 10⁻⁹ to 10⁻¹⁰ torr) tend to diffuse out into the residual gas atmosphere (typically 1 to 100 microns, although a GN₂ backfill in the mm range may also be used). The cleanup progress can be checked periodically by bleeding a sample through an external mass spectrometer through a heated pipe. If appropriate, the residual gas may be pumped off occasionally.

Outgassing at Transition Flow

In this method, the roughing pumps are used to continually remove residual gas, thus reducing readsorption of outgassed molecules, and speeding the cleanup. The penalty is some increased cost, for although these pumps may usually be left operating unattended, there is a cost involved in power and maintenance. An additional consideration is the possibility of backstreaming. This problem may be handled by careful trapping in the roughing line, or by a gas purge.

The gas purge might also be considered as a method of outgassing in itself. As discussed above, evaporants from low-vapor-pressure materials may be contained in a gas atmosphere, thus accelerating the net removal process as long as the mean free path remains large. One possible way to ensure gas flow sweeping behind shrouds and shields is to periodically vary the purge pressure over wide (10:1) limits. This would avoid high concentrations of contaminants in the small passage atmosphere. A problem to monitor with these methods is that the gas purge itself may introduce contaminants which limit the ultimate cleanliness.

A still faster variation of roughing pump outgassing is the addition of cryogenic panels within the chamber. These condense and concentrate contaminants for later removal by washing.

These transition flow methods are low in cost and effective in cleanup. For the very low-vapor-pressure contaminants, they appear to offer the most rapid cleanup, reducing the number of desorptions before pumping. The ultimate cleanliness may not be of quite as high a level as achieved with molecular flow outgassing. (The gas purge not only may introduce contaminants, but also interferes with their measurement.) Considering the contaminants introduced during installation of the test vehicle however, the net result may be the same.

Molecular Flow Outgassing

This method is similar to transition flow outgassing in continually removing contaminants. However, it uses a lower vacuum level and a different flow regime with different pumping speeds. Qualitatively, an outgassed molecule no longer diffuses out into surrounding gas, where there is a relatively high probability of being swept away. Instead, it travels directly to some other surface, where it recondenses.

In considering molecular flow, the concept of pumping fraction is useful. How many times must a molecule be desorbed before it is pumped? What is the probability of being pumped for an average desorption? For the typical space chamber this probability is very small, usually much less than 0.01. This means that the molecular flux within the chamber is essentially uniform over all surfaces - the reduction in flux near the pumps is negligible. This uniformity allows the pumping fractions to be calculated directly from the geometry: the effective area of the pump divided by total surface area.

Another useful concept is the Uniform Flux Theorem (Reference 9). Despite temperature differences of surfaces within the chamber, the molecular flux remains uniform. This means that heating only part of the surface (for example the shroud) simply concentrates material on the cool surfaces (in this example the chamber wall).

IMPROVED METHODS - ENERGIZING SURFACE, ENERGIZING MOLECULE, AND PREVENTIVE

Improved methods of cleanup are arbitrarily defined as those which require additional cost to implement. These methods are conceptually divided into three groups: methods which energize the surfaces, those which energize the contaminant molecules directly, and preventive methods.

Surfaces may be energized by shock and vibration; or by heating, as with pulsed radio frequency energy or lasers. Molecules may be directly energized by electron or ion bombardment, or ultraviolet radiation, or by chemical action, as with atomic oxygen. Preventive methods limit the amount and type of contaminant through selection of pump fluids, materials, and operations within the chamber.

Many of these accelerated methods are potentially harmful to painted or optical surfaces. However, some of them appear to be useful for specific limited applications.

SURFACE ENERGIZING METHODS - Shock, Vibration, and Gas Jets

It is commonly observed that mechanical shocks or vibration applied to a chamber will produce bursts of gas. However, tests showed only 20 percent particle removal by a 1,000-g, 1-msec shock of a particular surface under vacuum. (Reference 10.)

Gas jets, particularly after extended vacuum soak, were capable of removing up to 50 percent of the particles. These processes appear to be difficult to implement on a large scale. They do have some potential as a supplement to other methods. For example, a gas purge after extended vacuum soak might remove particles.

Heating

Short (microsecond) pulses of radio frequency energy have been used to heat thin (1 micron) layers at a surface to extreme temperatures (1,000°C) without heating the bulk material. (Reference 11.) Pulsed lasers have also been used to clean and erode surfaces by heating thin layers. (References 12 and 13.) Application of these methods to large areas presents difficulties in uniform heating. The maximum usable temperature is also limited by paint characteristics, reducing the potential effectiveness of these methods.

MOLECULE ENERGIZING METHODS - Electron Bombardment

Surfaces flooded by electrons release gas. This technique is widely used in the fabrication of vacuum tubes and in outgassing ionization gages. Equipment for this method is especially simple. Tungsten filaments are installed within the chamber. They are heated by adjustable currents and biased by adjustable voltages. The initial cleanup uses low voltages and currents to avoid arcing in the outgassed vapor. As the cleanup progresses, maximum available power is gradually applied. (Reference 14.) The electrons released from the filaments are drawn to the wall. On impact, they release secondary electrons, which tend to provide uniform flooding of all surfaces.

Two problems exist with application of this method to space chambers. Many contaminants will leave polymer films when electron bombarded; these could dull mirrors, or increase LN₂ consumption of shroud. Mayer has shown these films will form with electron energies as low as 6 volts. (Reference 15.) Painted surfaces may be damaged by electrons and, as insulators, may be difficult to bombard uniformly. However, there is at least the possibility that bombardment with low-energy electrons might be effective in removing contamination without damaging painted surfaces.

A very promising application is cleanup behind shrouds where there is usually no paint. This could be especially beneficial because contaminants tend to concentrate on the cool surfaces, as previously discussed.

Ion Bombardment

Surfaces bombarded by ions are eroded. This technique is widely used for cleaning substrates and for sputtering getter

material in ion pumps. Its use is recommended only for rugged, unpainted surfaces, because it is so destructive. Possible applications are for areas such as diffusion pump elbows and roughing line entrances.

Application is straightforward and only slightly more complex than electron bombardment. A simple approach is to use a combined static magnetic and electric field, as in ion pumps and magnetron gages. Radio frequency plasma is also common. Many geometries are available to fit specific situations. However, coverage is usually far from uniform, and contaminating deposits are often formed.

Ultraviolet Radiation

Ultraviolet radiation may directly energize a molecule; which may then either outgass, leaving the surface, or polymerize into a film. This method is attractive if a large solar beam is readily available. Rotating mirrors could illuminate most of the inner chamber surfaces. This method does degrade paints, but much more gradually than ion bombardment. A major problem is defining the level of contamination where formation of polymer films would be acceptable.

Chemical Action

Atomic oxygen rapidly combines with many materials. It is widely used in "dry ash" machines. It has been successfully used to remove polymer residues from mirrors (Reference 16.) Atomic oxygen from an RF plasma restored optical surfaces to their original properties by a rapid non-destructive oxidation of the polymer film. The test mirrors were cleaned within a 10-min time span, at a vacuum of 0.5 torr. Atomic oxygen also has been found to tend to restore white thermal coatings damaged by proton and ultraviolet radiation (Reference 17).

Application of this method to large space chambers presents interesting development problems. A low-temperature oxygen plasma may be generated by radio frequency, or by electron beam excitation. This plasma could be directed to flow over chamber surfaces, especially over solar optics. Other chemical reactions, such as with hydrogen, might also be used. Hazards in the use of these activated chemicals should be carefully considered.

PREVENTIVE METHODS

Pump Fluid

A new fluid for diffusion pumps and mechanical pumps (References 18, 19, and 20) offers the possibility of acceptable backstreaming incidents. The perfluoroalkyl polyether molecules do not adhere to surfaces and are easily removed. In addition,

they break up into completely volatile products when bombarded, leaving no residue.

This fluid requires more evaluation, but offers a potential solution to the serious problem of accidental backstreaming.

Porous Metal Screens

Porous metal screens have been suggested as a means of avoiding backstreaming during accidental repressurization.

Milleron (Reference 21) used a 1/16 inch thick stainless steel screen with 150 micron passages to isolate a diffusion pump. He found that the white vapor cloud formed by rapid repressurization was visually eliminated. However, quantitative measurements of backstreaming were not made. Screens could be used over both rough and diffusion pumps.

Material and Operation Selection

Lubricants for moving parts and for O-ring vacuum seals might be perfluoroalkyl polyether, similar to the pump fluid above. Electrical wiring might be insulated with ceramic; aluminum wire with aluminized oxide insulation is available. Test buildup operations can be arranged to minimize the work to be done in the chamber, and the time required for installation.

EVALUATION OF BASIC METHODS

What is the amount of contaminant a space vehicle will receive during a test, if the basic methods of cleanup are used to prepare the chamber? There is, of course, no simple general answer to this question. Yet, if we answer it as best we can, it will be helpful in considering the development of accelerated methods. Recent experience with the cleanup of S-1 leads to a tentative answer.

S-l is a 39-ft. diameter spherical chamber at the MDAC Huntington Beach Facility. It is roughed by mechanical pumps and blowers through the diffusion pumps. LN₂ cooled elbows (but no valves) separate the diffusion pumps from the chamber. On completion of a test series last fall (1972), the chamber was dirty, oily to the touch. It had been 3-1/2 years since the last major cleanup. During that time, 25 tests were conducted in 75 pumpdowns and 152 days under vacuum.

The cleanup procedure was a series of molecular flow outgassings. Prior to the first pumpdown, the chamber floor only was swept and wiped. The outgassing was done on weekends and holidays, while buildup for the next test continued during working days. Approximately 700 hours under vacuum were accumulated in seven pumpdowns. These included four consecutive weekends and one 10-day Christmas holiday. The last pumpdown followed a month of buildup, and installation of considerable

equipment, including a 190-ft² earth simulator, freshly painted with 3M Black Velvet 101ClO. Various combinations of shroud and building temperatures were used. (Figure 1.)

A number of interesting results emerged from this work. First, a residue of dry powder was left on the chamber surfaces. Despite the low outgassing, it later was necessary to wash this off.

The total pressures showed a substantial and cumulative improvement. (Figure 1.) The last pumpdown was especially surprising; even after a month of buildup and the large new outgassing surfaces, the improvement continued.

Most specific mass spectrometer (ms) peaks showed a rapid cleanup. One of the slowest was an unidentified material at AMU 350 (Figure 2). Note the dramatic response of this material to shroud heating. It finally disappeared below the threshold for good (10-13 torr for this particular instrument). Extrapolating the pressure decay transient, the present level must be much less than 1 monolayer/year.

(All mass spectrometer measurements presented are estimates based on nitrogen equivalent pressure. The ms viewed the center of S-1. The nature of the measurement is illustrated in Figure 3.)

Diffusion pump oil, DC-704 (AMU 199) cleaned up more rapidly than expected but showed some tendency to return on succeeding pumpdowns. It also is now below threshold. (Figure 4.)

Roughing pump oil, AMU 55 and 57, cleaned up very rapidly but was the only material that continued to return. (Figure 5.) A gas purge for use during diffusion pump shutdown and LN₂ baffle warmup is being installed. It is expected that this oil will also disappear below the threshold.

What do these results mean in terms of the original question, i.e., how much contaminant will a space vehicle receive? Pumpdown to 1 torr takes two hours in S-1; by this time, the shrouds may be cooled, ending contamination. During these two hours some part of the contaminant outgassing could reach the vehicle through turbulent flow. Conservatively, assume this fraction to be 1/10. If a specific contaminant is known to be outgassing at less than 1 monolayer/year, then during pumpdown less than 2.3×10^{-5} monolayers would be deposited on the vehicle. A similar amount might deposit during backfill. The total amount would be less than 10^{-4} monolayers for the particular contaminant.

This present limit is primarily one of measurement. If improved measurements could lead to assurance that the contamination rates were less than 1 monolayer/century, then the result would be below 10⁻⁶ monolayer. This appears to be an achievable goal.

The major contamination thus is expected to be that resulting from the installation of the particular vehicle.

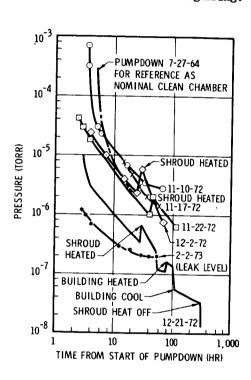
SUMMARY

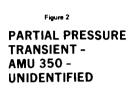
It has been experimentally demonstrated that the flux rates of various specific contaminants in a large (39 ft. diameter) space simulation chamber can be reduced to levels of less than 1 monolayer/year, with the cryogenic shrouds at room temperature. This corresponds to a vehicle contamination of less than 10-4 monolayers during the pumpdown-cooldown and warmup-backfill operations of a typical space simulation test. The cleaning procedures used were confined to washing and vacuum outgassing.

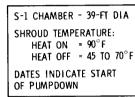
Figure 1
TOTAL PRESSURE
TRANSIENTS

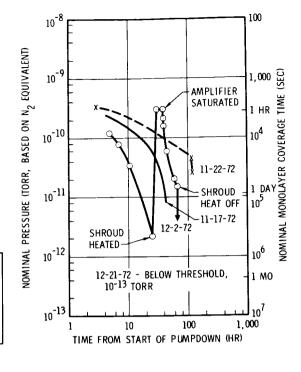
S-1 CHAMBER - 39-FT DIA
SHROUD TEMPERATURE:
HEAT ON = 90° F
HEAT OFF = 45 TO 70° F

DATES INDICATE START
OF PUMPDOWN









PUMPDOWN 2-3-73

T = 40 HR

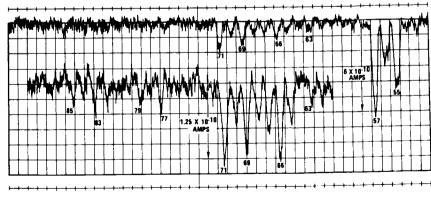
Figure 3

MASS SPECTROMETER SCAN
S-1 CHAMBER

20 AMP/TORR N2 EQUIVALENT

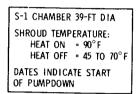
TWO SCANS AT DIFFERENT GAINS SUPERIMPOSED AMU 55 AND 57 ROUGHING PUMP OIL ,

OTHER PEAKS FROM FRESH PAINT AND OR HEATER TAPE OUTGASSING



Figure

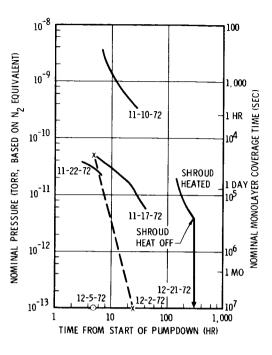
PARTIAL PRESSURE TRANSIENT -AMU 199 - DC 704

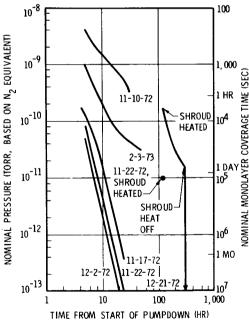


F:----- F

PARTIAL PRESSURE TRANSIENT - AMU 57 -ROUGHING PUMP OIL

S-1 CHAMBER - 39-FT DIA
SHROUD TEMPERATURE
HEAT ON = 90°F
HEAT OFF = 45 TO 70°F
DATES INDICATE START
OF PUMPDOWN





REFERENCES

- James T. Visentine, John W. Ogden, Melvin L. Ritter, Charles F. Smith, "Preparation, Verification, and Operational Control of Large Space-Environment-Simulation Chamber for Contamination Sensitive Tests." Proceedings of the Sixth Space Simulation Conference, p. 361 NASA SP-298, 1972.
- Philip W. Tashbar, Daniel B. Nisen, W. Walding Moore, Jr., "V-3 Contamination Test in the Manned Spacecraft Center's Vacuum Chamber-A." Proceedings of the Sixth Space Simulation Conference, p. 389 NASA SP-298, 1972.
- Dr. John J. Scialdone, "Predicting Spacecraft Self-Contamination in Space and in Test Chamber." Proceedings of the 6th Space Simulation Conference, p. 349 NASA SP-298, 1972
- 4. T. Baurer, M. H. Bortner, I. M. Pikus, A. M. Cooper, "External Spacecraft Contamination Modeling and Countermeasures." Proceedings of the Fifth Space Simulation Conference, p. 79 NBS SP-337, 1970.
- J. C. Goldsmith, E. R. Nelson, "Molecular Contamination in Environmental Testing at Goddard Space Flight Center." Proceedings of the Fifth Space Simulation Conference, p. 1 NBS SP-336, 1970.
- L. Holland, "Review of Some Recent Vacuum Studies." Vacuum, v. 20, p. 175, 1970.
- 7. C. Burch, "Some Experiments on Vacuum Distillation," Royal Society London Proc. Seriea A, V. 123, p. 271-287, 1929.
- J. H. de Boer, "The Dynamical Character of Adsorption." Clarendon Press, Oxford, 1953.
- 9. B. C. Moore, "Measurement of Vacuum in Non-uniform Temperature Environments," Journal of Vacuum Science & Technology, 1, p. 10-16, 1964.
- 10. D. L. Enlow, "Contamination Studies in a Space Simulated Environment." Proceedings of the Fifth Space Simulation Conference, p. 51 NBS SP-336, 1970.
- K. I. Lobacher, V. A. Shiskin, USSR Patent No. 263, 752, appl. 17 Jan 1968, Abstract Vacuum, 21, 287, 1971.
- S. M. Bedair, H. P. Smith, Jr., "Atomically Clean Surfaces by Pulsed Laser Bombardment," J. Appl. Phys., 40, 4776-81, 1969.

- 13. L. P. Levine, J. F. Ready and E. Bernal G, "Laser Bombard-ment Effects on Vacuum Surfaces," Research/Development Dec. 1965, p. 56-59.
- 14. R. E. Clausing, "Release of Gases from Surfaces by Energetic Electrons," (paper presented at the Eleventh National Vacuum Symposium, Chicago, Illinois, Sept. 30-Oct. 2, 1964).
- 15. L. Mayer, "Photocontrol of Growth Rate of Thin Polymer Films Formed by Electron Bombardment," J. Applied Physics, 34, 2088-2093.
- R. E. Gillette, J. R. Hoilahan, G. L. Carlson, "Restoration of Optical Properties of Surfaces by Radio Frequency-Excited Oxygen," J. Vac. Sci. & Tech., 7, 534-537, 1970.
- 17. R. E. Gillette and W. D. Beverly, "Restoration of Degraded Spacecraft Surfaces Using Reactive Gas Plasmas," AIAA Paper No. 71-463, AIAA 6th Thermal Physics Conference, Tullahoma, Tenn., April 26-28, 1971.
- 18. M. A. Baker, L. Holland, L. Laurenson, "The Use of Perfluoroalkyl Polyether Fluids in Vacuum Pumps," Vacuum, v. 21, p. 479-81, 1971 (Oct).
- L. Holland, "The Use of Perfluoroalkyl Polyether Lubricants in Mechanically Driven Vacuum Pumps," Vacuum, v. 22, p. 234 1972 (June).
- L. Holland, L. Laurenson, P. W. Baker, "The Use of Perfluoropolyether Fluids in Vapour Stream Pumps," Vacuum, 22, p. 315-319, August 1972.
- N. Millerson, "Porous Metal Isolation Traps and Cryosorbents in Vacuum Technique," 1965 Transactions of the Third International Vacuum Congress, v. 2, p. 189-192.
 H. Adams editor, Pergamon Press, NY, 1967.